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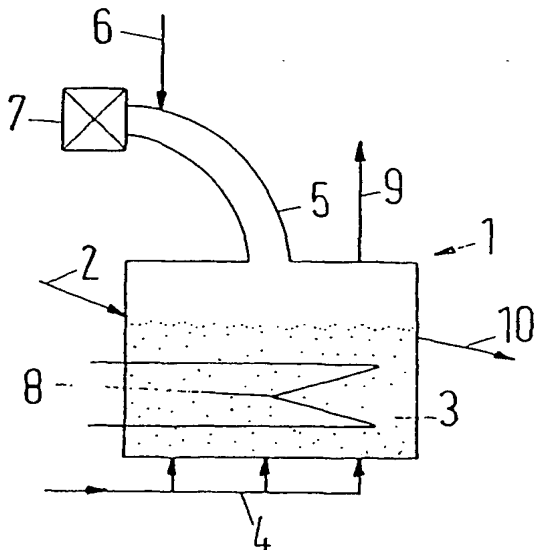
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**(54) Title:** METHOD AND PLANT FOR THE THERMAL TREATMENT OF GRANULAR SOLIDS IN A FLUIDIZED BED



**(57) Abstract:** This invention relates to a method for the thermal treatment of granular solids in a fluidized bed (3, 3a) which is located in a fluidized-bed reactor (1, 1a, 38), wherein microwave radiation is fed into the fluidized-bed reactor (1, 1a, 38) through at least one wave guide (5, 46), and to a corresponding plant. To avoid depositions in the wave guide (5, 46), a gas stream is fed into the fluidized-bed reactor (1, 1a, 38) through the same wave guide (5, 46).

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## METHOD AND PLANT FOR THE THERMAL TREATMENT OF GRANULAR SOLIDS IN A FLUIDIZED BED

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### Technical Field

( 10 This invention relates to a method for the thermal treatment of granular solids in a fluidized bed which is located in a fluidized-bed reactor, wherein microwave radiation is fed into the fluidized-bed reactor through at least one wave guide, and to a corresponding plant.

15 There are several possibilities for coupling a microwave source to fluidized-bed reactors. These include for instance an open wave guide, a slot antenna, a coupling loop, a diaphragm, a coaxial antenna filled with gas or another dielectric, or a wave guide occluded with a microwave-transparent substance (window). The type of decoupling the microwaves from the feed conduit can be effected in different ways.

- 20 Theoretically, microwave energy can be transported in wave guides free of loss. The wave guide cross-section is obtained as a logical development of an electric oscillating circuit comprising coil and capacitor towards very high frequencies. Theoretically, such oscillating circuit can likewise be operated free of loss. In the case of a substantial increase of the resonance frequency, the coil of an electric oscillating circuit becomes half a winding, which corresponds to the one side of the wave guide cross-section. The capacitor becomes a plate capacitor, which likewise corresponds to two sides of the wave guide cross-section. In reality, an oscillating circuit loses energy due to the ohmic resistance in coil and capacitor. The wave guide loses energy due to the ohmic resistance in the wave guide wall.

30 Energy can be branched off from an electric oscillating circuit by coupling a second oscillating circuit thereto, which withdraws energy from the first one. Similarly, by flanging a second wave guide to a first wave guide energy can be decoupled from the same (wave guide transition). When the first wave guide is shut off behind the coupling point by a shorting plunger, the entire energy can even be diverted to the second wave guide.

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The microwave energy in a wave guide is enclosed by the electrically conductive walls. In the walls, wall currents are flowing, and in the wave guide cross-section an electromagnetic field exists, whose field strength can be several 10 KV per meter.

5 When an electrically conductive antenna rod is put into the wave guide, the same can directly dissipate the potential difference of the electromagnetic field and with a suitable shape also emit the same again at its end (antenna or probe decoupling). An antenna rod which enters the wave guide through an opening and contacts the wave guide wall at another point can still directly receive wall currents and likewise emit the same at its

10 end. When the wave guide is shut off by a shorting plunger behind the antenna coupling, the entire energy can be diverted from the wave guide into the antenna in this case as well.

When the field lines of the wall currents in wave guides are interrupted by slots,

15 microwave energy emerges from the wave guide through these slots (slot decoupling), as the energy cannot flow on in the wall. The wall currents in a rectangular wave guide flow parallel to the center line on the middle of the broad side of the wave guide, and transverse to the center line on the middle of the narrow side of the wave guide. Transverse slots in the broad side and longitudinal slots in the narrow side therefore

20 decouple microwave radiation from wave guides.

Microwave radiation can be conducted in electrically conductive hollow sections of all kinds of geometries, as long as their dimensions do not fall below certain minimum values. The exact calculation of the resonance conditions involves rather complex

25 mathematics, as the Maxwell equations (unsteady, nonlinear differential equations) must ultimately be solved with the corresponding marginal conditions. In the case of a rectangular or round wave guide cross-section, however, the equations can be simplified to such an extent that they can be solved analytically and problems as regards the design of wave guides become clearer and are easier to solve. Therefore,

30 and due to the relatively easy producibility, only rectangular wave guides or round wave guides are used industrially, which are also preferably used in accordance with the invention. The chiefly used rectangular wave guides are standardized in the Anglo-Saxon literature. These standard dimensions were adopted in Germany, which is why odd dimensions appear in part. In general, all industrial microwave sources of the

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frequency 2.45 GHz are equipped with a rectangular wave guide of the typ R26, which has a cross-section of 43 x 86 mm. In wave guides, different oscillation states exist: In the transversal electric mode (TE mode), the electric field component lies transverse to the wave guide direction and the magnetic component lies in wave guide direction. In the transversal magnetic mode (TM mode), the magnetic field component lies transverse to the wave guide direction and the electric component lies in wave guide direction. Both oscillation states can appear in all directions in space with different mode numbers (e.g. TE-1-1, TM-2-0).

A method for the thermal treatment of granular solids is known from US 5,972,302, wherein sulfidic ore is subjected to an oxidation supported by microwaves. This method is chiefly concerned with the calcination of pyrite in a fluidized bed, wherein the microwaves introduced into the fluidized bed promote the formation of hematite and elementary sulfur and suppress the formation of SO<sub>2</sub>. There is employed a stationary fluidized bed which is directly irradiated by the microwave source disposed directly above the same. The microwave source or the entrance point of the microwaves necessarily gets in contact with the gases, vapors and dusts ascending from the fluidized bed.

EP 0 403 820 B1 describes a method for drying substances in a fluidized bed, wherein the microwave source is disposed outside the fluidized bed and the microwaves are introduced into the fluidized bed by means of a wave guide. Open wave guides involve the risk that the microwave source is soiled by dust and gases and damaged in the course of time. This can be avoided by microwave-transparent windows, which occlude the wave guide between the reactor and the microwave source. In this case, however, deposits on the window lead to an impairment of the microwave radiation.

### Description of the Invention

It is therefore the object underlying the invention to make the feeding of microwaves into a stationary or circulating fluidized bed more efficient and protect the microwave source against resulting gases, vapors and/or dusts.

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In accordance with the invention, this object is substantially solved in a method as mentioned above in that a gas stream is fed into the fluidized-bed reactor through the wave guide, which is also used for introducing microwaves. Thus, the microwave source is disposed outside the stationary or circulating fluidized bed, the microwave radiation being fed into the fluidized-bed reactor through at least one wave guide and a gas stream being passed through the wave guide in addition to the microwave radiation. By means of the gas stream from the wave guide it is reliably avoided that dust or process gases enter the wave guide, spread up to the microwave source and damage the same. In accordance with the invention, microwave-transparent windows in the wave guide for shielding the microwave source, as they are commonly used in the prior art, can therefore be omitted. The same involve the problem that deposits of dust or other solids on the window can impair and partly absorb the microwave radiation. Therefore, the open wave guides in accordance with the invention are particularly advantageous.

An improvement of the method is achieved when the gas stream introduced through the wave guide contains gases which react with the fluidized bed and in the case of a circulating fluidized-bed reactor can even be utilized for an additional fluidization of the fluidized bed. Thus, part of the gas which so far has been introduced into the fluidized bed through other supply conduits is used for dedusting the wave guide. As a result, providing neutral purge gas can also be omitted.

Another improvement is obtained in accordance with the invention when the gas stream introduced through the wave guide has a temperature difference with respect to the gases and solids present in the fluidized-bed reactor. In this way, additional heat can specifically be introduced into the fluidized bed or the fluidized bed can be cooled, depending on the desired effect.

The thermal treatment can not only be effected in a stationary, but also in a circulating fluidized bed, wherein the solids circulate continuously between a fluidized-bed reactor, a solids separator connected with the upper region of the fluidized-bed reactor and a return conduit connecting the solids separator with the lower region of the fluidized-bed reactor. Usually, the amount of solids circulating per hour is at least three times the amount of solids present in the fluidized-bed reactor.

The solids can also be passed through at least two succeeding fluidized-bed reactors, for instance two fluidization chambers separated from each other by means of weirs or partitions, in which the stationary fluidized beds are formed and to which the electromagnetic waves (microwaves) coming from wave guides are fed. The solids can move from one fluidized-bed reactor into the adjacent fluidized-bed reactor. One variant consists in that between the two adjacent fluidized-bed reactors an intermediate chamber is disposed, which is in particular connected with both fluidization chambers and contains a fluidized bed of granular solids, the intermediate chamber having no associated wave guide. Another variant of the method of the invention consists in that a partition with the opening in the bottom region is used for separating the two fluidization chambers.

To a particular advantage, the operating conditions, in particular temperature, composition of the fluidizing gas, energy input and/or fluidization rate can be specified differently for each of several fluidized-bed reactors. In the case of one fluidized bed or several succeeding fluidized beds, the solids thus can for instance first be passed through a preheating chamber upstream of the first fluidized bed. Furthermore, downstream of the last fluidized bed serving the thermal treatment a cooling chamber may be provided for cooling the solid product.

Another advantage is obtained in that solid deposits in the wave guide are avoided by the continuous gas stream through the wave guide. These solid deposits change the cross-section of the wave guide in an undesired way and absorb part of the microwave energy which was designed for the solids in the fluidized bed. Due to the absorption of energy in the wave guide, the same is heated very much, whereby the material is subject to a strong thermal wear. In addition, solid deposits in the wave guide effect undesired feedbacks to the microwave source.

Suitable microwave sources, i.e. sources for the electromagnetic waves, include e.g. a magnetron or a klystron. Furthermore, high-frequency generators with corresponding coils or power transistors can be used. The frequencies of the electromagnetic waves proceeding from the microwave source usually lie in the range from 300 MHz to 30 GHz. Preferably, the ISM frequencies 435 MHz, 915 MHz and 2.45 GHz are used.

Expediently, the optimum frequencies are determined for each application in a trial operation.

5 In accordance with the invention, the wave guide wholly or largely consists of electrically conductive material, e.g. copper. The length of the wave guide lies in the range from 0.1 to 10 m. The wave guide may be straight or curved. There are preferably used sections of round or rectangular cross-section, the dimensions being in particular adapted to the frequency used.

10 The temperatures in the fluidized bed lie for instance in the range from 300 to 1200°C, and it may be recommended to introduce additional heat into the fluidized bed, e.g. through indirect heat transfer. For temperature measurement in the fluidized bed, insulated sensing elements, radiation pyrometers or fiber-optic sensors can be used.

15 In accordance with the invention, the gas velocities in the wave guide are adjusted such that the Particle-Froude-Numbers in the wave guide lie in the range between 0.1 and 100. The Particle-Froude-Numbers are defined as follows:

$$Fr_p = \frac{u}{\sqrt{\frac{(\rho_s - \rho_f)}{\rho_f} * d_p * g}}$$

20 with

u = effective velocity of the gas flow in m/s

$\rho_s$  = density of the solid particles or process gases penetrating into the wave guide in kg/m<sup>3</sup>

25  $\rho_f$  = effective density of the purge gas in kg/m<sup>3</sup>

$d_p$  = mean diameter in m of the particles of the reactor inventory (or the particles formed) during operation of the reactor

g = gravitational constant in m/s<sup>2</sup>.

30 To prevent solid particles or generated process gases from the reactor from penetrating into the wave guide, gas serving as purge gas flows through the wave guide. Solid



particles can for instance be dust particles present in the reactor or also the treated solids. Process gases are generated in the processes which take place in the reactor. By specifying certain Particle-Froude-Numbers, the density ratio of the penetrating solid particles or process gases to the purge gas is considered in accordance with the invention when adjusting the gas velocities, which ratio, apart from the velocity of the  
5 purge gas stream, is decisive for the question whether or not the purge gas stream can entrain the penetrating particles. Substances can thereby be prevented from penetrating into the wave guide. For most applications, a Particle-Froude-Number between 2 and 30 is preferred.

10 The granular solids to be treated by the method in accordance with the invention can for instance be ores and in particular sulfidic ores, which are prepared e.g. for recovering gold, copper or zinc. Furthermore, recycling substances, e.g. zinc-containing processing oxide or waste substances, can be subjected to the thermal treatment in the  
15 fluidized bed. If sulfidic ores, such as e.g. auriferous arsenopyrite, are subjected to the method, the sulfide is converted to oxide, and with a suitable procedure there is preferably formed elementary sulfur and only small amounts of  $\text{SO}_2$ . The method of the invention favorably loosens the structure of the ore, so that the subsequent gold leaching leads to improved yields. The arsenic iron sulfide ( $\text{FeAsS}$ ) preferably formed  
20 by the thermal treatment can easily be disposed of. Expediently, the solids to be treated at least partly absorb the electromagnetic radiation used and thus heat the bed. It was surprisingly found out that in particular material treated at high field strengths can be leached more easily. Frequently, other technical advantages can be realized as well, such as reduced retention times or a decrease of the required process  
25 temperatures.

The present invention furthermore relates to a plant in particular for performing the above-described method for the thermal treatment of granular solids in a fluidized bed. A plant in accordance with the invention includes a fluidized-bed reactor, a microwave  
30 source disposed outside the fluidized-bed reactor, and a wave guide for feeding the microwave radiation into the fluidized-bed reactor, a gas supply conduit for feeding gas into the fluidized-bed reactor being connected to the wave guide.

Furthermore, the reactor can be elongated and have a gas-permeable bottom for the entrance of fluidizing gas, for instance a bottom provided with hole or slot openings, bell nozzles or similar openings suitable for fluidization technology. This reactor designed as fluidized-bed channel can be installed horizontally or with a small angle of inclination of a few degrees and have a length/width ratio of at least 1.5 to 1, for instance 4 to 1. In such a reactor, the treatment and the transport of the granular solids can easily be realized in accordance with the invention. To divide the fluidization channel reactor in several zones, partitions or weirs can be arranged inside the fluidized bed formed in the channel and/or in the gas space located above the fluidized bed, depending on the process, an opening being left for the passage of the granular solids. It is particularly advantageous when the partitions or weirs are adjustable for separating zones, so that the height of the fluidizing material and the slot height can be varied for the transfer from zone to zone. The bed depth in the fluidization channel is selected such that in each zone an almost uniform energy state is achieved due to a thorough mixing. In the case of a suitable fluidizing material, the siphon principle can also be used instead of transfer weirs. Microwave inlet openings with wave guides connected thereto can be provided in all zones or in individual zones.

Developments, advantages and possibilities for applying the present invention can also be taken from the following description of examples and from the drawing. All described and/or illustrated features per se or in any combination belong to the subject-matter of the invention, independent of their inclusion in the claims or their back-reference.

### Brief Description of the Drawings

In the drawings

Fig. 1 shows the thermal treatment of granular solids in a stationary fluidized bed in a schematic representation;

Fig. 2 shows a method variant with a circulating fluidized bed; and

Figs. 3, 4, 5, 6 show method variants with a plurality of stationary fluidized beds.

### Detailed Description of the Preferred Embodiments

Fig. 1 shows a plant for performing the method in accordance with the invention for the thermal treatment of granular solids in a stationary fluidized layer 3 which is also referred to as fluidized bed.

The plant includes a fluidized-bed reactor 1, into which granular solids to be treated are introduced through a conduit 2. In a chamber, the solids form a stationary fluidized bed 3 which is traversed by a fluidizing gas, e.g. air. For this purpose, the fluidizing gas is passed from below through a gas distributor 4 into the fluidized bed 3. In the upper region of the fluidized-bed reactor 1, an open wave guide 5, which leads to a microwave source 7, is connected to the chamber with the stationary fluidized bed 3. The electromagnetic waves proceeding from the microwave source 7 are passed through the wave guide 5 and fed into the chamber of the fluidized-bed reactor 1. They at least partly contribute to the heating of the fluidized bed 3. Furthermore, purge gas, e.g. air or nitrogen, is laterally fed into the wave guide 5 through a conduit 6, which purge gas flows into the fluidized-bed reactor 1 and prevents the ingress of dust or process gases from the chamber with the fluidized bed 3 into the wave guide 5. In this way, the microwave source 7 is protected against being damaged, and at the same time microwave-absorbing soil deposits in the wave guide 5 are prevented without the open wave guide 5 having to be closed by a window transparent for microwaves.

If necessary for the process, the stationary fluidized bed 3 can additionally be heated by a heat exchanger 8 disposed in the fluidized bed 3. Gases and vapors formed leave the chamber of the fluidized-bed reactor 1 through a conduit 9 and are supplied to a non-illustrated cooling and dedusting known per se. The treated granular solids are withdrawn from the fluidized-bed reactor 1 through the discharge conduit 10.

In Fig. 2, the fluidized-bed reactor 1 constitutes a reactor with a circulating fluidized bed (fluidized layer). The solids to be treated are introduced into the fluidized-bed reactor 1 via conduit 2 and entrained by fluidizing gas introduced into the fluidized-bed reactor 1, whereby the circulating fluidized layer is formed. The solids then are at least partly discharged from the fluidized-bed reactor 1 along with the gas through a conduit 11 and introduced into a solids separator 12. The solids separated therein are at least partly

recirculated through a return conduit 13 into the lower region of the circulating fluidized layer of the fluidized-bed reactor 1. Part of the solids can also be discharged through the discharge conduit 14. Coarse solids, which are deposited at the bottom of the fluidized-bed reactor 1, can be removed from the reactor 1 through a discharge conduit 15. The fluidizing gas for forming the circulating fluidized bed, e.g. air, is supplied to the fluidized-bed reactor 1 through a conduit 4a and then first gets into a distribution chamber 4h, before it flows into the fluidized-bed reactor 1 through a grid 4i, entrains the introduced, in particular fine-grained solids and forms a circulating fluidized layer as fluidized bed.

A wave guide 5 connects a microwave source 7 with the chamber of the fluidized-bed reactor 1, through which wave guide microwaves are fed into the microwave reactor 1 for heating the granular solids as in the plant in accordance with Fig. 1. In addition, purge gas from conduit 6 flows through the wave guide 5, in order to avoid the ingress of dirt as well as deposits in the wave guide 5. In the present case as well, the inner region of the chamber can again be provided with one or more heat exchangers for additionally heating the granular solids, which for a better clarity was not represented in Fig. 2.

Dust-laden gas leaves the solids separator 12 through conduit 9 and is first cooled in a waste heat boiler 16, before it is passed through a dedusting 17. Separated dust can either be removed from the process or be recirculated to the chamber of the fluidized-bed reactor 1 through a non-illustrated conduit.

As shown in Fig. 3, two stationary fluidized-bed reactors 1 and 1a are arranged in series, an intermediate chamber 1c being located between the chambers of the two reactors 1 and 1a. In all three chambers, the solids form a stationary fluidized bed 3, 3a, which is traversed by fluidizing gas. The fluidizing gas for each chamber is supplied through a separate conduit 4a, 4b, 4c, respectively. The granular solids to be treated enter the first fluidized-bed reactor 1 through conduit 2, and completely treated solids leave the second fluidized-bed reactor 1a through the discharge conduit 10. From the upper region of the chamber of the first reactor 1 a first wall 19 extends downwards. However, it does not extend down to the ground, so that in the bottom region an opening 20 is left, through which solids from the first fluidized bed 3 can get into the

fluidized bed 3a of the intermediate chamber 1c. The intermediate chamber 1c extends up to a weir-like second wall 21, over which the solids from the fluidized bed 3a of the intermediate chamber 1c are moved into the chamber of the second fluidized-bed reactor 1a. Corresponding to the plants as shown in Figs. 1 and 2, wave guides 5 with  
5 purge air conduits 6 and microwave sources 7 are each connected to the chambers of the two reactors 1 and 1a, through which wave guides the microwaves and purge gas are fed into the reactors 1 and 1a. In the chambers of the reactors 1 and 1a, heat exchanging elements 8 may be arranged in addition.

( )  
10 The gas space 22 above the fluidized bed 3 of the first fluidized-bed reactor 1 is separated from the gas space 23, which belongs to the chamber of the second reactor 1a and the intermediate chamber 1c, by the vertical wall 19. For the gas spaces 22, 23 separate gas discharge conduits 9 and 9a exist. As a result, different conditions can be maintained in the chambers of the reactors 1 and 1a, in particular different  
15 temperatures can exist or different fluidizing gases can be supplied through separate gas supply conduits 4a, 4b, 4c. Furthermore, the two microwave sources 7 can be designed differently and perform different functions. In particular, microwaves of different frequency or energy can be generated and be introduced through the wave  
guide 5.

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( ) As shown in Fig. 4, two stationary fluidized-bed reactors 1 and 1a without intermediate chamber are arranged directly succeeding each other, a partition 19 being disposed between the two. In the chambers of the two reactors 1, 1a the solids form a stationary fluidized bed 3, 3a, which is fluidized by fluidizing gas from several conduits 4a, 4b, 4c  
25 disposed one beside the other. The granular solids to be treated are supplied to the first fluidized-bed reactor 1 through conduit 2, and the treated solids leave the fluidized-bed reactor 1a through the discharge conduit 10. From the upper region of the chamber of the first reactor 1, a first wall 19 extends downwards, which does, however, not extend down to the ground, so that in the bottom region an opening 20 is left, through  
30 which solids from the first fluidized bed 3 can get into the fluidized bed 3a of the second fluidized-bed reactor 1a. Waveguides 5, which are connected to the microwave sources 7, each extend to the two chambers of the reactors 1 and 1a. According to the principle described already in the previous embodiments, microwaves are fed into the two reactors 1, 1a through these open wave guides 5, in order to heat the solids to be

treated, which absorb the microwave radiation, and reach the necessary process temperatures. At the same time, purge gas flows into the wave guides 5 through purge air conduits 6, in order to avoid deposits in the same. In the chambers of the reactors 1 and 1a, heat exchanging elements 8 may be arranged in addition.

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The gas space 22 above the fluidized bed 3 of the first fluidized-bed reactor 1 is separated from the gas space 23, which belongs to the chamber of the second reactor 1a, by the vertical wall 19. There exist separate gas discharge conduits 9 and 9a. As a result, different conditions can be maintained in the different reactor chambers 1 and 1a; in particular, the temperatures or the gas phase composition can be different. Different fluidizing gases can also be supplied through the respective conduits 4a, 4b, 4c. Furthermore, the two microwave sources 7 can be designed differently and perform different functions.

10

15 In the arrangement as shown in Fig. 5, the solids to be treated, which are supplied via conduit 2, first enter an antechamber 31 and flow through a first intermediate chamber 32 in the first fluidized-bed reactor 1. The solids then are discharged from the same to flow through a second intermediate chamber 1c into the second fluidized-bed reactor 1a and finally through the third intermediate chamber 33 into a cooling chamber 34, before the treated and cooled solids are withdrawn through the discharge conduit 10. Wave guides 5 with associated non-illustrated microwave sources each open into the chambers of the fluidized-bed reactors 1 and 1a, in order to feed microwaves into the reactors 1 and 1a according to the above-described principle. All chambers include stationary fluidized beds, to which fluidizing gas is supplied through separate gas supply conduits 4a to 4g for each chamber. The exhaust gases are discharged through corresponding conduits 9a to 9d.

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In the cooling chamber 34, the fluidized bed includes a cooling means 35 for an indirect heat transfer, whose cooling fluid, e.g. cooling water, is heated in the cooling means 35 and then supplied through conduit 36 to the heat exchanger 37 in the preheating chamber 31. There, the cooling fluid releases part of its heat to the solids in the fluidized bed disposed there, whereby a very economic utilization of heat is achieved.

30

As variant of another plant in accordance with the invention, Fig. 6 shows a fluidization channel reactor 38, in which the fluidized layer is formed in a channel-type bottom 39 with through openings for a fluidizing gas. The fluidization channel reactor 38 is divided into four zones 41a to 41d separated by adjustable partitions 40, the first zone 41a constituting a preheating zone, the second zone 41b an oxidation zone, the third zone 41c a reduction zone, and the fourth zone 41d a cooling zone. Downstream of each of the zones 41a to 41d a separator 42 or a cyclone is provided, which separates the solids discharged with the fluidizing gas from the gas stream and recirculates the same to the respective zone 41a to 41d. To achieve a high utilization of energy, the exhaust gases from the separators 42 are supplied to other zones 41a to 41d by means of a suitable gas recirculation.

Via a feed conduit 43, the solids to be treated are supplied to the first zone 41a of the reactor 38. As fluidizing gas, hot exhaust gas from a first combustion chamber 44 is supplied to the first zone 41a, in order to dry and preheat the introduced material. The correspondingly preheated solids flow through the partition 40 into the oxidation zone 41b, to which there is likewise supplied hot exhaust gas from a second combustion chamber 45. To both combustion chambers 44, 45, supply conduits are connected for fuel and air and possibly preheated exhaust gas from other process zones 41a to 41d. From the oxidation zone 41b, the solids are supplied to the reduction zone 41c. For protecting the downstream compressor, the exhaust gas from the oxidation zone 41b can likewise be supplied to the reduction zone 41c via a cooler 47. Possibly, the exhaust gas is again heated in a heater 49.

To bring the fluidized material to the desired energy state, microwave rays are additionally introduced into the oxidation zone 41a and the reduction zone 41c through wave guides 46 traversed by purge gas. Due to the microwave radiation, the solids are heated by an internal excitation, so that the energy state can easily be adjusted. In the last zone 41d, the treated material is cooled with injected air and discharged as product through the discharge conduit 48.

To make the feeding of microwaves into a stationary or circulating fluidized bed 3, 3a more efficient and also protect the microwave source 7 against the resulting gases, vapors and dusts, the microwave source 7 in accordance with the invention is disposed

outside the stationary or circulating fluidized bed 3, 3a and the fluidized-bed reactors 1, 1a, 38. The microwave radiation is fed into the fluidized-bed reactor 1, 1a, 38 through at least one open wave guide 5, 46, wherein in addition to the microwave radiation a gas stream flows into the fluidized-bed reactor 1, 1a, 38 through the wave guide 5, 46. By means of the gas stream, the wave guide 5, 46 is kept dust-free, which considerably increases the efficiency of the introduction of microwaves.

#### **Example 1 (Calcination of ores containing pyrite)**

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Pyrite with grain sizes in the range from 80 to 160  $\mu\text{m}$  is treated in two successive fluidized beds 3, 3a, which are designed corresponding to the plant in accordance with Fig. 4. Irradiation is effected in both chambers by microwaves with a frequency of 2.45 GHz. As radiation source, magnetrons are used.

15

Into the first fluidized-bed reactor 1, 182.5 kg/h pyrite are charged. For fluidizing the fluidized bed 3, 360  $\text{Nm}^3/\text{h}$  nitrogen are used, which are supplied through conduit 4a, so that a height of 20 cm is obtained for the fluidized bed. After the microwave treatment, the mass flow rate of the solid reaction products from the first fluidized-bed reactor 1 is 153.5 kg/h. The first chamber is operated at 550°C and a magnetron irradiation of 36 kW.

20

Deoiled compressed air with a volume flow rate of 120  $\text{Nm}^3/\text{h}$  is supplied to the second fluidized bed 3a through conduit 4c. The second chamber is operated at 500°C and a microwave irradiation of 36 kW. After 90 min, a steady state is obtained; after the microwave treatment, the mass flow rate of the solid reaction products is 140.2 kg/h.

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As purge gas, there is each utilized the gas used for fluidization, i.e. in the first chamber nitrogen and in the second chamber deoiled compressed air, which each have a volume flow rate of 50  $\text{Nm}^3/\text{h}$ .

30

The phase content of the pyrite used and of the products of the first and second process stages is analysed by X-ray diffraction. In the pyrite, only  $\text{FeS}_2$  can be detected. After the first temperature treatment, the solids consist of substoichiometric



FeS and FeS<sub>2</sub> for instance in accordance with FeS<sub>x</sub> with  $x = 1.4$ . After the second stage, no more sulfur-containing products can be detected, the solids virtually exclusively consist of hematite.

## 5 Example 2 (Calcination of ore containing gold)

On a laboratory scale, gold ore with grain sizes in the range below 250 µm is treated in a circulating fluidized bed which is designed as shown in Fig. 2. Irradiation is effected by microwaves with a frequency of 2.45 GHz. As radiation source, a magnetron is used. For purging, 24 Nm<sup>3</sup>/h air are supplied to the reactor 1 through the wave guide 5.

### Feed

Type gold ore, ground, dried and classified

### Grain fraction

15 Max µm 250

Composition Wt-%

Org. C 1.05

CaCO<sub>3</sub> 19.3

20 Al<sub>2</sub>O<sub>3</sub> 12.44

FeS<sub>2</sub> 2.75

Inerts, e.g. SiO<sub>2</sub> 64.46

Input, about kg 100

### 25 Apparatus

Type of reactor circulating fluidized bed with microwave irradiation

Reactor diameter mm 200

Magnetron 500 W, 2.45 GHz

Microwave coupling wave guide, R26 (43 x 86 mm) designed as secondary air conduit

30 Connected: online gas analysis + exhaust gas washing

Operation: continuous

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## Test conditions and results

|    |   |                    | Inlet | Outlet |
|----|---|--------------------|-------|--------|
|    | Mass flow rate, gold ore feed   | kg/h               | 195   |        |
|    | Primary air   | °C                 | 250   |        |
| 5  |   | Nm <sup>3</sup> /h | 30    |        |
|    |   | °C                 | 50    |        |
|    | Oil consumption   | kg/h               | 0.70  |        |
|    | Secondary air,  |                    |       |        |
|    | preheated by means of Luvo to   | °C                 | 425   |        |
| 10 | Secondary air, consumption  | Nm <sup>3</sup> /h | 24    |        |
|    | Drier air   | °C                 | 50    | 320    |
|    |   | Nm <sup>3</sup> /h | 70    | 70     |
|    | Calcining residue, ex-WS luvo   | °C                 |       | 400    |
|    |   | kg/h               |       | 182    |
| 15 | Calcining gas, total  |                    |       |        |
|    |   | Nm <sup>3</sup> /h |       | 59     |
|    |   | °C                 |       | 600    |
|    | Composition, dry  |                    |       |        |
| 20 | CO <sub>2</sub>   | vol-%              |       | 23.3   |
|    | N <sub>2</sub>  | vol-%              |       | 74.3   |
|    | O <sub>2</sub>  | vol-%              |       | 2.4    |
|    | SO <sub>2</sub>   | ppV                |       | 134.1  |
| 25 | The phase content of the material used and of the products is analysed by X-ray diffraction. After the treatment, neither residual sulfur nor organic carbon can be detected in the calcining residue, the solids have a pale gray color. |                    |       |        |

**List of Reference Numerals:**

|    |         |                                |          |                              |
|----|---------|--------------------------------|----------|------------------------------|
|    | 1,1a    | fluidized-bed reactor          | 20       | opening                      |
|    | 1c      | intermediate chamber           | 21       | weir, partition              |
| 5  | 2       | conduit                        | 31       | antechamber                  |
|    | 3,3a    | fluidized layer, fluidized bed | 32       | intermediate chamber         |
|    | 4       | gas distributor                | 33       | intermediate chamber         |
|    | 4a to g | conduits                       | 34       | cooling chamber              |
|    | 4h      | distribution chamber           | 35       | cooling means                |
| 10 | 4i      | grid                           | 36       | conduit                      |
|    | 5       | wave guide                     | 37       | heat exchanger               |
|    | 6       | conduit                        | 38       | fluidization channel reactor |
|    | 7       | microwave source               | 39       | bottom                       |
|    | 8       | heat exchanger                 | 40       | partitions                   |
| 15 | 9       | conduit, gas discharge conduit | 41a to d | zones                        |
|    | 10      | discharge conduit              | 42       | separator                    |
|    | 11      | conduit                        | 43       | feed conduit                 |
|    | 12      | solids separator               | 44       | combustion chamber           |
|    | 13      | return conduit                 | 45       | combustion chamber           |
| 20 | 14      | discharge conduit              | 46       | wave guide                   |
|    | 15      | discharge conduit              | 47       | cooler                       |
|    | 16      | waste heat boiler              | 48       | discharge conduit            |
|    | 17      | dedusting                      | 49       | heater                       |
| 25 | 19      | weir, partition                |          |                              |

## Claims

1. A method for the thermal treatment of granular solids in a fluidized bed (3, 3a) which is located in a fluidized-bed reactor (1, 1a, 38), wherein microwave radiation is fed into the fluidized-bed reactor (1, 1a, 38) through at least one wave guide (5, 46),  
5 **characterized in** that a gas stream is fed into the fluidized-bed reactor (1, 1a, 38) through the same wave guide (5, 46).
2. The method as claimed in claim 1, **characterized in** that the gas stream introduced through the wave guide (5, 46) contains gases which react with the fluidized  
10 bed (3, 3a).
3. The method as claimed in claim 1 or 2, **characterized in** that the gas stream introduced through the wave guide (5, 46) is additionally utilized for a fluidization of the  
15 fluidized bed (3, 3a).
4. The method as claimed in any of the preceding claims, **characterized in** that heat is additionally supplied to the fluidized bed (3, 3a) through the introduced gas stream.  
20
5. The method as claimed in any of claims 1 to 3, **characterized in** that the fluidized bed (3, 3a) is cooled by the introduced gas stream.
6. The method as claimed in any of the preceding claims, **characterized in** that  
25 the fluidized bed (3, 3a) is part of a stationary and/or circulating fluidized bed.
7. The method as claimed in any of the preceding claims, **characterized in** that the reactor comprises at least two fluidized-bed reactors (1, 1a, 41a to d), which are separated from each other by weirs or partitions (19, 21, 40) such that solids can move  
30 as moving bed from one fluidized-bed reactor (1, 41a to c) into an adjacent fluidized-bed reactor (1a, 41b to d).
8. The method as claimed in claim 7, **characterized in** that the operating conditions, in particular temperature, composition of the fluidizing gas, energy input

and/or fluidization rate, can be specified differently for each fluidized-bed reactor (1, 1a, 41a to d).

9. The method as claimed in any of the preceding claims, **characterized in** that by means of the gas stream introduced into the wave guide (5, 46) solid deposits in the wave guide (5, 46) are avoided.

10. The method as claimed in any of the preceding claims, **characterized in** that the used frequency of the microwave radiation lies between 300 MHz and 30 GHz, preferably at the frequencies 435 MHz, 915 MHz and 2.45 GHz.

11. The method as claimed in any of the preceding claims, **characterized in** that the temperatures in the fluidized bed (3, 3a) lie between 300°C and 1200°C.

12. The method as claimed in any of the preceding claims, **characterized in** that the Particle-Froude-Number  $Fr_p$  in the wave guide (5, 46) is 0.1 to 100, preferably 2 to 30.

13. A plant for the thermal treatment of granular solids in a fluidized bed (3, 3a), in particular for performing the method as claimed in any of claims 1 to 12, comprising a fluidized-bed reactor (1, 1a, 38), a microwave source (7) disposed outside the fluidized-bed reactor (1, 1a, 38) and a wave guide (5, 46) for feeding the microwave radiation into the fluidized-bed reactor (1), **characterized in** that a gas supply conduit (6) is connected to the wave guide (5, 46) for feeding gas into the fluidized-bed reactor (1, 1a, 38).

14. The plant as claimed in claim 13, **characterized in** that the wave guide (5) has a rectangular or round cross-section, whose dimensions are adapted in particular to the used frequency of the microwave radiation.

15. The plant as claimed in claim 13 or 14, **characterized in** that the wave guide (5, 46) has a length of 0.1 m to 10 m.

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Fig.1

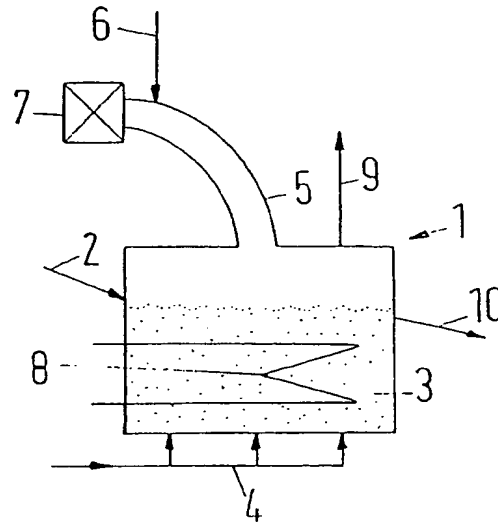
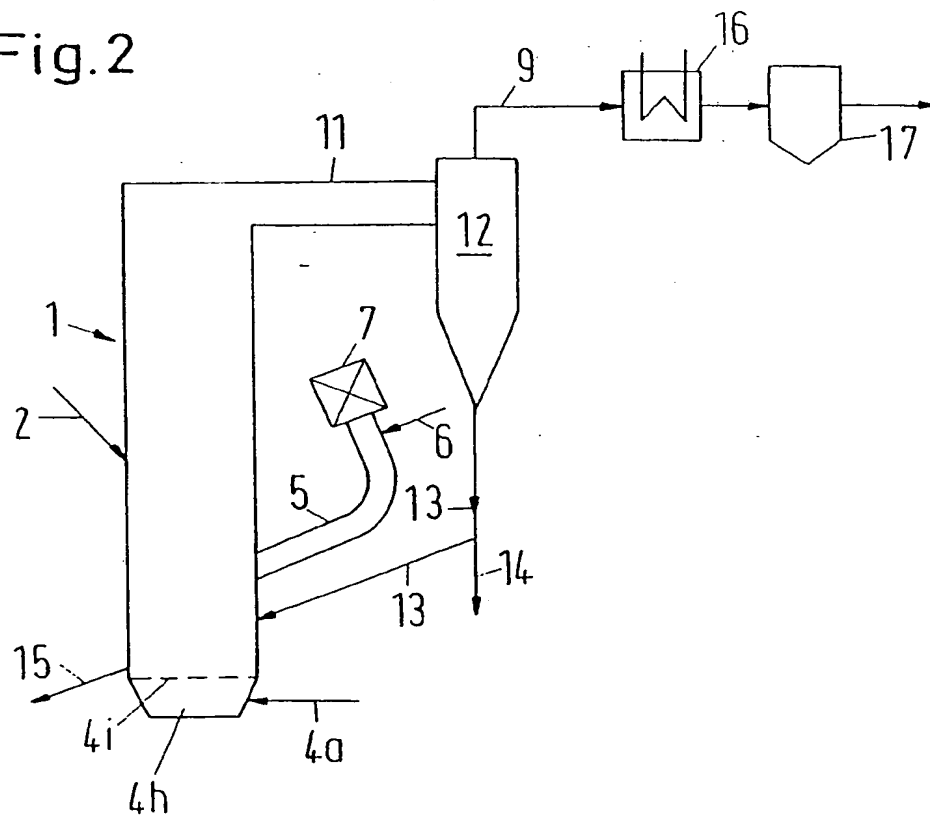


Fig.2



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Fig.3

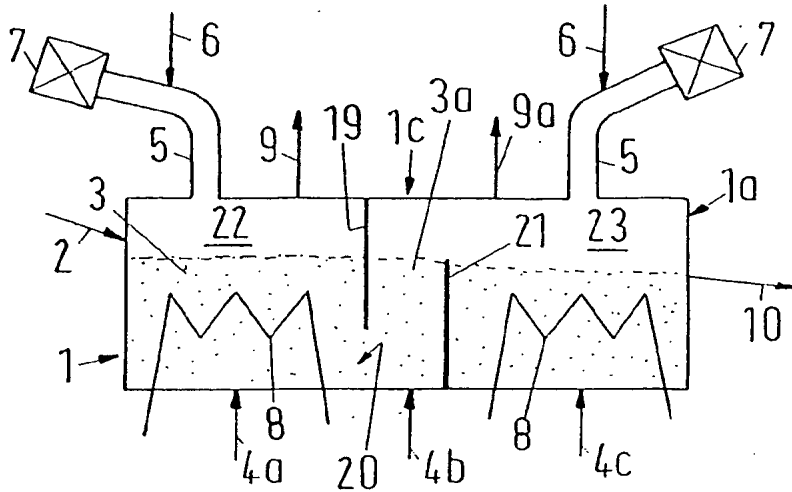


Fig.4

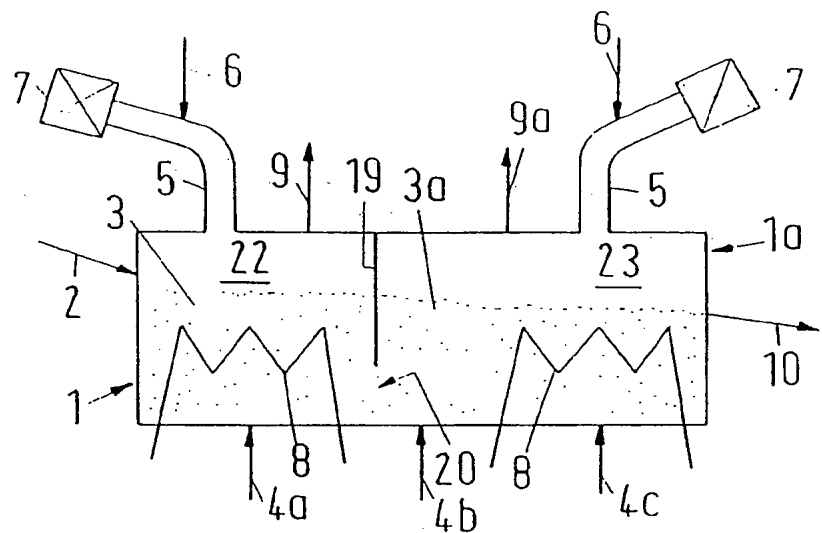
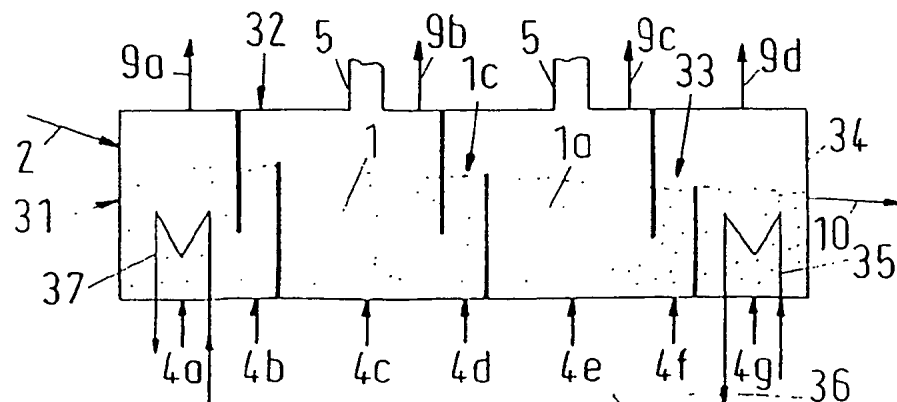
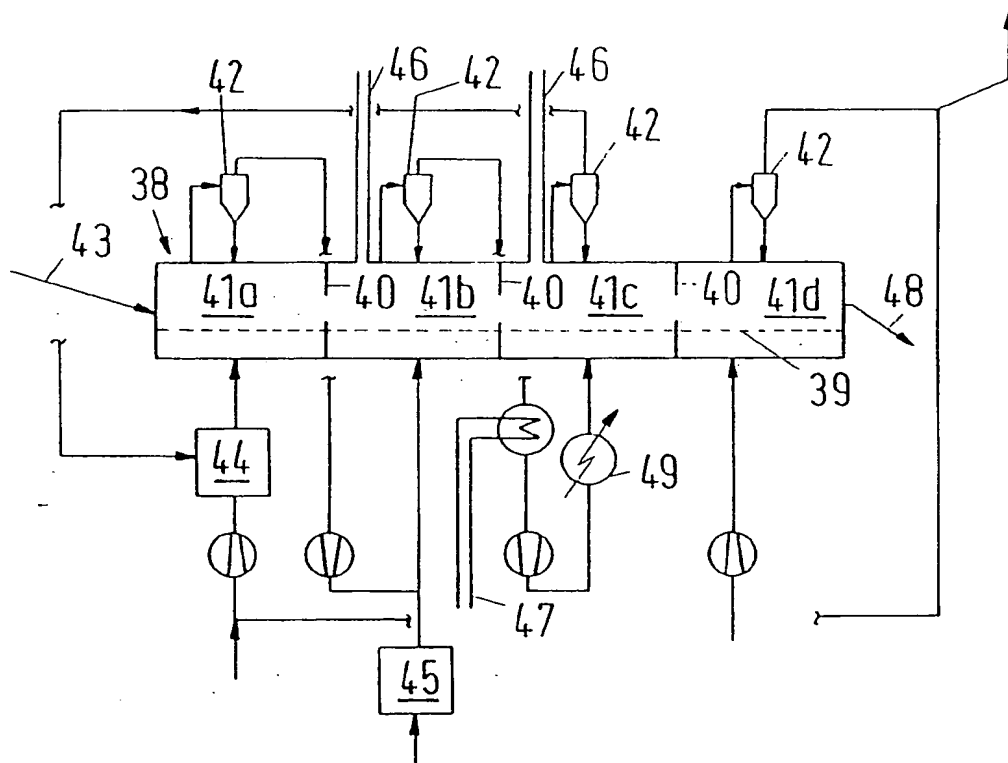


Fig.5



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